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DETERMINATION OF THE HIGH-TEMPERATURE STRENGTH OF CERAMIC OXIDE MATERIALS

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The results of a determination of the high-temperature (to 1500°C) compression strength (for light-weight fiber blocks) and bending strength (dense ceramic oxide) are presented. The measurements are performed on an Instron 5882 universal setup equipped with a resistance furnace.

Key words: compression strength, bending strength, fiber blocks, ceramic oxide, mullite.

The use of mullite-based fiber tiles and ceramic elements as high-temperature filters and thermal insulation for high-temperature furnaces and in other technological applications requires a thorough study of their physical properties at high temperatures, since all calculations of part design are based on these properties [1, 2].

High-temperature tests of materials based on oxide fibers and dense ceramic oxide were performed on an Instron 5882 setup together with a resistance furnace with working temperature from 800 to 1500°C and outfitted with alundum pushers and equipment, made of SiC, for performing compression and four-point bending experiments in air [3]. The temperature regime was set by a programmer; two thermocouples were place in the hot zone: controlling and monitoring, placed in direct proximity to the sample. Heating to the required temperature was done at a rate 8 – 12 K/min. The samples were soaked at this temperature, during which time mechanical experiments were performed. The experimental data — the loading and deformation of a sample (during bending tests) — were displayed on a computer monitor.

The compression strength of rigid fiber material (10% linear deformation) and the strength of dense mullite ceramic samples under four-point bending determined in the experiments are presented below. The duration of a single experiment, including heating and cooling, was 5 – 6 h. The thermal expansion of the parts placed in the hot zone of the furnace had to be taken into account; these were mainly alundum pushers, largely because of their length. Calculations of the thermal expansion of the pushers showed that to a first approximation it equals 4 mm at 1300°C with total length about 500 mm. The thermal expansion of the SiC equipment and the sample itself can be neglected, on the basis of which (to prevent spontaneous loading) the sample was moved at last 4 mm away from the pusher. Once the prescribed temperature was reached (immediately before the tests began) the sample was placed in contact with the pusher, which was indicated by a sharp increase of the load displayed on the monitor screen.

The samples cut from fiber blanks with the minimum spread of the density and strength values at room temperature were chosen for the high-temperature experiments designed to determine the compression strength with 10% deformation.

Determination of the Temperature Dependence of the Compression Strength with 10% Linear Deformation of a Rigid Material Made of Discrete Fibers

The stress arising under compression (in what follows, the strength) was chosen as a criterion for evaluating the strength of a material.

Material No. 1. The density of this material is in the range 0.45 – 0.46 g/cm^3. The strength at room temperature and at 1400 and 1500°C is shown in Table 1.

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TABLE 1. Strength of Materials with Different Density versus the Temperature

Material	Density, g/cm^3	Compression strength,* MPa, at temperature, °C					
		20	1000	1200	1300	1400	1500
No. 1	0.45 – 0.46	0.30 ± 0.06	–	–	–	0.31	0.19 ± 0.01
No. 2	0.40 – 0.01	0.30 ± 0.03	–	0.27	0.20	0.21	0.10 ± 0.01
No. 3	0.31 ± 0.01	0.25 ± 0.02	0.17	0.14	0.14	0.14	0.07

* Strength with 10% linear deformation of a material.

It can be concluded from the results of high-temperature tests that the strength levels for this material decrease very little right down to 1400°C. As temperature rises above this level the strength drops sharply.

Material No. 2. The room-temperature tests (see Table 1) gave average strength 0.30 ± 0.03 MPa with material density 0.40 ± 0.01 g/cm^3. The high-temperature strength at 1400°C is less than the corresponding values obtained for material No. 1, probably because of the more than 10% lower density of material No. 2. In addition, the strength of material No. 2 started to decrease at 1200°C.

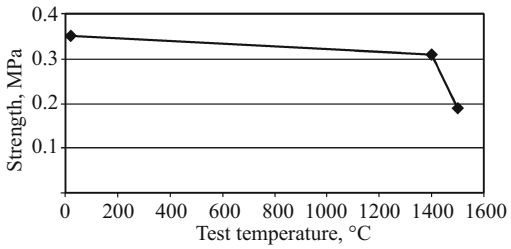

Fig. 1. Compression strength of fiber material No. 1 versus the test temperature.

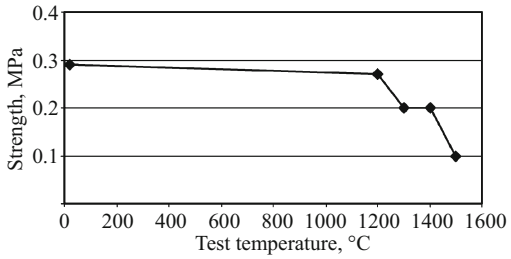

Fig. 2. High-temperature compression strength of material No. 2 versus the test temperature.

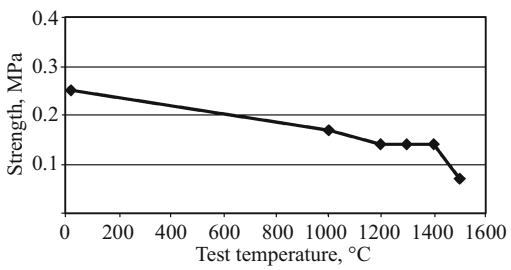

Fig. 3. High-temperature compression strength of fiber material No. 3 versus the test temperature.

The change in the strength of the material with increasing test temperature is presented in Fig. 2.

The density decrease for this material at 1500°C is larger than for material No. 1.

Material No. 3. The density of this material is 0.31 ± 0.01 g/cm^3. The strength values determined at different test temperatures are presented in Table 1. The strength at 1300°C was 0.14 MPa, which is 1.5 – 1.8 times lower than the values obtained at room temperature. A noticeable drop in strength starts at 1000°C. We recall that in material No. 1 the strength at 1400°C is practically the same as at room temperature.

The strength of material No. 3 at 1500°C equals 0.07 MPa. This is 2.7 times lower than for material No. 1 at the given temperature and 1.4 times lower than for material No. 2.

The change in strength for material No. 3 with increasing test temperature is presented in Fig. 3.

Analysis. The experiments showed that the high-temperature compression strength with 10% linear deformation of rigid material based on discrete fibers is proportional to the density of the material (Fig. 4).

It was found that the temperature at which the strength of rigid material based on discrete fibers drops by 30% is proportional to the density of the material (Fig. 5).

Determination of the High-Temperature (1300°C) Strength of Ceramic Material Based on Mullite ($3Al_2O_3 \cdot 2SiO_2$) Under Four-Point Bending

A series of four-point bending tests (40 mm base with 20 mm separation of the top supports) performed on ceramic material (90% mullite + 10% zirconium oxide) was conducted at room temperature and 1300°C. The technological history of the samples was the same. The density of the samples was 3.2 g/cm^3; the surface was worked by polishing with a free abrasive on a cast iron plate; the average size of the last fraction used was 24 μm.

The test results obtained at room temperature and at 1300°C are presented in Table 2.

TABLE 2. Strength of Mullite-Based Ceramic Material at 20 and 1300°C

Sample No.	Bending strength, MPa, at temperature, °C	
	20	1300
1	230	110
2	280	150
3	160	160
4	210	170
5	230	180

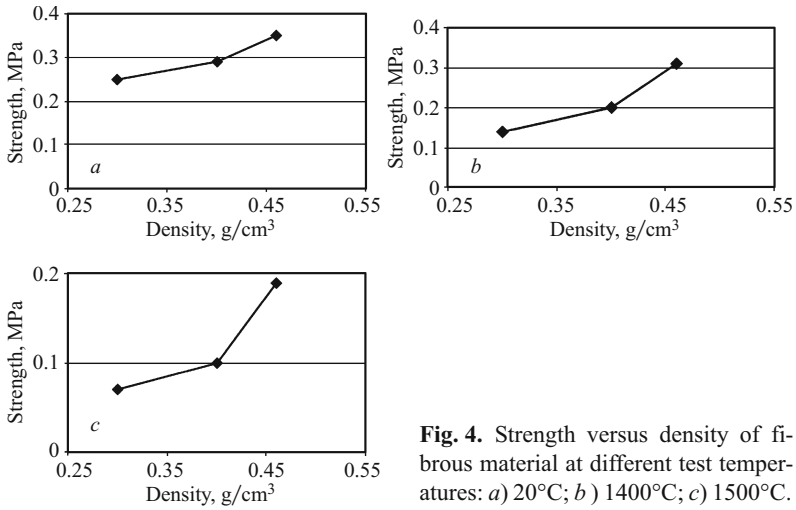

Fig. 4. Strength versus density of fibrous material at different test temperatures: *a*) 20°C; *b*) 1400°C; *c*) 1500°C.

The high-temperature tests performed with an Instron 5882 setup equipped with a resistance furnace gave, against all expectations, lower strength values at 1300°C than at room temperature. This can be explained by the fact that the test temperature was somewhat higher than optimal for the given composition of the material. Apparently, for mullite materials containing zirconium oxide the temperature of maximum strength is somewhat lower than the value given in most works for pure mullite (1300 – 1400°C), and lies in the range 1200 – 1250°C. It can be supposed that physically the drop in the temperature of maximum strength is due to the development of grain slippage, close to the mechanism of superplasticity, similar to the processes occurring in the system Al_2O_3–ZrO_2 and in pure zirconium oxide [4].

In addition, the high-temperature (1300°C) plastic deformation of the sample during four-point bending under a load, equal to about 50% of the maximum strength, was investigated. In the course of the experiment the sample was loaded to approximately 200 N (20 kgf), and a drop in the load due to the plastic deformation of the sample was compensated manually using a precision positioning setup on the control panel of the Instron 5882 setup.

The experimental results are presented in Fig. 6. The experimental curve shows that prior to creep there is an incubation period lasting longer than 1 h, after which plastic deformation starts and proceeds, to a first approximation, at a constant rate. The residual sag of the sample after 2-h tests was 0.5 mm on a 4 mm base.

The result obtained confirms that at 1300°C mullite containing zirconium oxide additives possesses substantial plasticity, the measured deformation arising after a sufficiently long incubation period and developing at an approximately constant rate. In short-time tests with loading rate 2 mm/min this effect was not seen and only decreased the measured value of the strength.

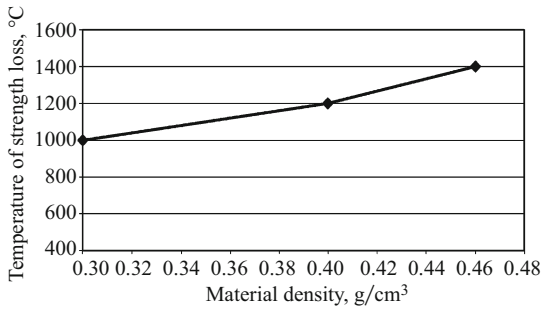

Fig. 5. Temperature of 30% strength loss of rigid material based on discrete fibers versus the density of the material.

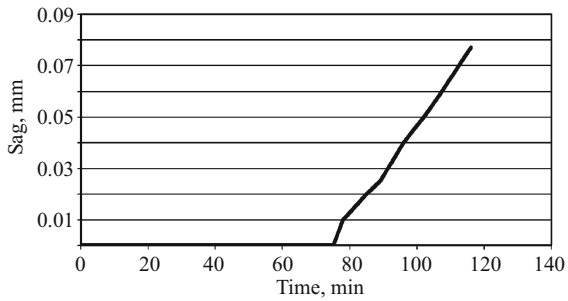

Fig. 6. Sag increase with four-point bending under a constant about 200 N (20 kgf) at 1300°C in time.

CONCLUSIONS

Methods for performing high-temperature tests performed on materials with 10% compression and four-point bending were tested using an Instron 5882 setup equipped with a high-temperature furnace.

High-Temperature Compression Tests on Rigid Material Consisting of Discrete Fibers with 10% Linear Deformation

The tests showed that the high-temperature compression strength is proportional to the density of the rigid material based on discrete fibers. It was also found that the tempera-

ture threshold for 30% strength loss of this material is proportional to its density.

High-Temperature Four-Point Bending Tests on Mullite Ceramic

The temperature of maximum bending strength of the experimental mullite materials containing zirconium oxide is in the range 1200 – 1250°C. As temperature increases to 1300°C the short-time bending strength decreases by a small amount. Thus, the temperature at which the strength decreases is appreciably lower than for pure mullite. The experiment studying the behavior of such materials at 1300°C showed that under a load equal to 50% of the maximum strength at this temperature plastic deformation of the sample starts after an incubation period of the order of 1 h and then proceeds at a constant rate.

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